1 381 588

PATENT SPECIFICATION (11) 1 381 588

(21) Application No. 43187/73 (22) Filed 14 Sept. 1973
(31) Convention Application No. 2 246 013 (32) Filed 20 Sept. 1972 in (19)
(33) Germany (DT)
(44) Complete Specification published 22 Jan. 1975
(51) INT CL² A61J 3/10
(52) Index at acceptance A5B 750 75Y 764

(54) PROCESS FOR THE PREPARATION OF POROUS TABLETS

(71) We, BOEHRINGER MANNHEIM G.M.B.H., of Mannheim-Waldhof, Federal Republic of Germany, a Body Corporate organised under the laws of the Federal Republic of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention is concerned with a new process for the preparation of porous tablets.

Because of the ease of handling and the simplicity of dosing, not only pharmaceutical tablets but also reagent tablets are used to an ever increasing extent for diagnostic and analytical purposes. Most active materials and reagents cannot be tabletted by themselves since they [illegible] The present invention is concerned with a [illegible] behind [illegible] are damaged by solvents and readily brea [illegible] cassary cases, tend t [illegible] ction

Tablets v [illegible] esent obtained b [illegible] rmits agents, such [illegible] orous or the like, [illegible] thout phosphates [illegible] its or as talc, st [illegible] Whereas it [illegible] ntion, logically c [illegible] ction ceuticals, [illegible] com- speaking, [illegible] which break down quickly are only cannot be produced or can only be produced [illegible] of the tablets. [illegible] hard with difficulty in this manner. In particular, the lubricants which are generally used and which are intended to prevent the adherence of the tablet masses in the presses used are mostly insoluble in water. It has, therefore, been suggested to press together adhesive reagents with very large amounts of readily tablettable fillers or to use very high pressures for the pressing. However, both processes are unsatisfactory since the tablets formed are either unnecessary large or are too hard and difficult to break down.

Another known process gives so-called "moulded tablets". In this case, the tablet components are pasted with water or an organic solvent, in which at least one of the components partially dissolves, to give a stiff slurry which is formed in special machines to give tablets, whereafter the tablets are carefully dried. Upon evaporation of the solvent, the substances dissolved therein stick together the undissolved particles, whereby the tablets receive their strength; at the same time, small hollow spaces remain behind into which the solvents can again penetrate upon dissolving again. Although these tablets are satisfactory from the point of value of speed of dissolving, they are frequently too soft and brittle due to the presence of the very fine canals so that difficulties arise with packing and transport. Furthermore, the use of the process is limited due to the fact that many reagents, especially enzymes and

ERRATA

SPECIFICATION No. 1,381,588

Page 3, line 57, *for* nitotinamide *read* nicotinamide

Page 4, Table 3, right hand column, bottom line, *for* 15 *read* |<15

Page 4, line 7, *for* 116 mg. *read* 11.6 mg.

THE PATENT OFFICE
24th March, 1975

pressed together with at least one inert solid adjuvant, which sublimes at a temperature which does not adversely affect any of the tablet components, whereafter said adjuvant is sublimated.

The tablet components are to be understood to mean all those components, other than the sublimable adjuvant, which constitute the tablet, such as active materials and phamcaceutical carriers and diluents.

Due to the hard pressing in conventional tabletting machines, there are formed tablets of great mechanical stability and, at the same time, the addition of sparingly soluble lubricants is unnecessary. Since the pressed

SEE ERRATA SLIP ATTACHED

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The present invention is concerned with a new process for the preparation of porous tablets.

Because of the ease of handling and the simplicity of dosing, not only pharmaceutical tablets but also reagent tablets are used to an ever increasing extent for diagnostic and analytical purposes. Most active materials and reagents cannot be tabletted by themselves since they form hard tablets which do not readily break down and, in addition, in many cases, tend to stick in the presses used.

Tablets which break down quickly are only obtained by the addition of disintegration agents, such as carboxymethyl-cellulose, starch or the like, filling materials, such as lactose, phosphates and the like, and lubricants, such as talc, stearic acid, paraffin or the like. Whereas it is simple to find suitable physiologically compatible adjuvants for pharmaceuticals, reagent tablets which, generally speaking, are to give optically clear solutions, cannot be produced or can only be produced with difficulty in this manner. In particular, the lubricants which are generally used and which are intended to prevent the adherence of the tablet masses in the presses used are mostly insoluble in water. It has, therefore, been suggested to press together adhesive reagents with very large amounts of readily tablettable fillers or to use very high pressures for the pressing. However, both processes are unsatisfactory since the tablets formed are either unnecessary large or are too hard and difficult to break down.

Another known process gives so-called "moulded tablets". In this case, the tablet components are pasted with water or an organic solvent, in which at least one of the components partially dissolves, to give a stiff slurry which is formed in special machines to give tablets, whereafter the tablets are carefully dried. Upon evaporation of the solvent, the substances dissolved therein stick together the undissolved particles, whereby the tablets receive their strength; at the same time, small hollow spaces remain behind into which the solvents can again penetrate upon dissolving again. Although these tablets are satisfactory from the point of value of speed of dissolving, they are frequently too soft and brittle due to the presence of the very fine canals so that difficulties arise with packing and transport. Furthermore, the use of the process is limited due to the fact that many reagents, especially enzymes and indicators, are damaged by solvents and organic solvent vapours make necessary special safety requirements in the production of the tablets.

It is, therefore, an object of the present invention to provide a process which permits the production of readily dissolved, porous tablets in conventional tablet presses, without having to add lubricants, explosive agents or solvents.

Thus, according to the present invention, there is provided a process for the production of porous tablets, wherein the tablet components as hereinafter defined are hard pressed together with at least one inert solid adjuvant, which sublimes at a temperature which does not adversely affect any of the tablet components, whereafter said adjuvant is sublimated.

The tablet components are to be understood to mean all those components, other than the sublimable adjuvant, which constitute the tablet, such as active materials and phamcaceutical carriers and diluents.

Due to the hard pressing in conventional tabletting machines, there are formed tablets of great mechanical stability and, at the same time, the addition of sparingly soluble lubricants is unnecessary. Since the pressed

tablets, in contradistinction to the "moulded tablets", are form-stable, they no longer shrink upon removal of the adjuvant. Therefore, when the adjuvant is removed, it leaves behind comparatively large hollow spaces and canals, through which solvent can penetrate.

As adjuvants, there can be used, in principle, all readily sublimable materials or materials which can readily be converted into gaseous decomposition products and which are readily tablettable and do not react with the other components of the tablets. By way of example, there may be mentioned urethane, urea, ammonium carbonate and bicarbonate, hexamethylene-tetramine, benzoic acid, phthalic anhydride, naphthalene and camphor, urethane being specially preferred.

The tablet masses for water-soluble reagent tablets and pharmaceutical tablets can, in addition to one or more active materials, contain conventional water soluble carrier materials, for example sodium chloride, potassium chloride, borax, phosphates, oligosaccharides, polyethylene glycols, tensides and other appropriate inorganic and organic materials. The volatile solid adjuvants can account for 5—50% by weight and preferably 10—30% of the total tablet mass, it being understood that in the case of a high proportion of adjuvant, there are formed comparatively large hollow spaces and thus tablets which break down more quickly but are also more brittle than in the case of using a small proportion of adjuvant. Although the adjuvants can be completely removed, the production time for the new tablets according to the present invention is shortened when the adjuvants are allowed to remain behind in the tablets in trace amounts, for example of less than 1% by weight.

In the case of sufficient thermal stability, the adjuvants can be removed by simple heating of the tablets above the sublimation or decomposition point. In the case of sensitive tablet components, for example of enzymes, it is advantageous to work in a vacuum, the conventional freeze drying plants with condensation separator having proved to be especially advantageous for this purpose.

The following Examples are given for the purpose of illustrating the present invention:—

Example 1.

Tablet A: 1.850 kg. potassium chloride are sieved and pressed to form tablets of 8 mm. diameter containing 185 mg. potassium chloride.

Tablet B1: 1.850 kg. potassium chloride are mixed with 350 g. urethane (ethylurethane), sieved and pressed to form tablets of 8 mm. diameter containing 185 mg. potassium chloride and 35 mg. urethane.

The urethane is subsequently sublimated off from these tablets for 5 hours in a freeze drying plant at 20°C. and at a pressure of 10^{-1} to 10^{-3} mm.Hg.

Tablet B2: 1.850 kg. potassium chloride are mixed with 350 g. ammonium bicarbonate, sieved and pressed to form tablets of 8 mm. diameter containing 185 mg. potassium chloride and 35 mg. ammonium bicarbonate.

The ammonium bicarbonate is driven off from these tablets for 8 hours in a drying cabinet at 90°C.

Tablet B3: 1.850 kg. potassium chloride are mixed with 350 g. urea, sieved and pressed to form tablets of 8 mm. diameter containing 185 mg. potassium chloride and 35 mg. urea.

The urea is sublimated off from these tablets for 16 hours in a vacuum cabinet at 110°C. and 15 mm.Hg.

Tablet B4: 1.850 kg. potassium chloride are mixed with 350 g. urotropin, sieved and pressed to form tablets of 8 mm. diameter containing 185 mg. potassium chloride and 35 mg. urotropin.

The urotropin is removed from these tablets for 16 hours in a vacuum cobinet at 90°C. and 15 mm.Hg.

The results of tests carried out on these tablets are set out in the following Table 1:—

TABLE 1

tablet	height (mm.)	hardness (kg.)	dissolving time (sec.)	breakability (sec.)
A	2.3	9.5	240	150
B1 – B4	2.9	3.5	105	30

Determination of the tablet hardness: with a Erweka hardness tester.

Determination of the dissolving time: 200 ml. water at ambient temperature are stirred at a rate of 150 r.p.m. in a 250 ml. glass beaker with a curved glass rod. The time needed for complete dissolving is determined.

Determination of breakability: a tablet placed on its edge in a Petri dish is compressed

with a rod with an applied weight of 500 g. The Petri dish is filled with water at ambient temperature and the time determined for the tablet to break.

Example 2.

Tablet A: 1.5 kg. dextrose are granulated with 40% alcohol, dried and sieved. The granulate is dry mixed with 50 g. polyethylene glycol (M.W. 5000—6000) and pressed to form tablets of 8 mm. diameter containing 150 mg. dextrose.

Tablet B1: 1.550 kg. dextrose-polyethylene glycol granulate are dry mixed with 300 g. urethane. The tablet mass is pressed to form tablets of 8 mm. dia meter containing 150 mg. dextrose and 30 mg. urethane.

The urethane is sublimated from these tablets for 8 hours in a drying cabinet at 40°C.

Tablet B2: 1.550 kg. dextrose-polyethylene glycol granulate are dry mixed with 300 g. ammonium carbonate. The tablet mass is pressed to form tablets of 8 mm. diameter containing 150 mg. dextrose and 30 mg. ammonium carbonate.

The ammonium carbonate is removed from these tablets for 8 hours in a drying cabinet at 75°C.

Tablet B3: 1.550 kg. dextrose-polyethylene glycol granulate are dry mixed with 300 g. benzoic acid. The tablet mass is pressed to form tablets of 8 mm. diameter containing 150 mg. dextrose and 30 mg. benzoic acid.

The benzoic acid is sublimated from these tablets for 16 hours in a vacuum cabinet at 90°C. and 15 mm.Hg.

Tablet B4: 1.550 kg. dextrose-polyethylene glycol granulate are dry mixed with 300 g. camphor. The tablet mass is pressed to form tablets of 8 mm. diameter containing 150 mg. dextrose and 300 mg. camphor.

The camphor is removed from these tablets for 8 hours in a freeze drying device at 40°C. and 10^{-1} to 10^{-3} mm.Hg.

The results of tests carried out on these tablets, in the manner described in Example 1, are set out in the following Table 2:—

TABLE 2

tablet	height (mm.)	hardness (kg.)	dissolving time (sec.)	breakability (sec.)
A	2.7	4.5	360	210
B1 – B4	3.3	1.0	270	<10

Example 3.

Tablet A: 15 g. polyethylene glycol (M.W. 5000—6000) are dissolved in 40% alcohol. With this solution, there are granulated 388 g. glucose and 12.5 g. nitotinamide - adenine - dinucleotide (NAD), 3.75 g. 2,5 - diphenyl - 3 - (4,5-dimethyl - thiazolyl - 2) - tetrazolium bromide (MTT) and 0.75 g. phenazine methosulphate (PMS) are added thereto. The mixture is pressed to form tablets of 12 mm. diameter, each tablet containing 12.5 mg. NAD, 3.75 mg. MTT and 0.75 mg. PMS.

Tablet B: 15 g. polyethylene glycol (M.W. 5000—6000) are dissolved in 40% alcohol. With this solution, there are granulated 388 g. glucose, which is then dried and sieved. The granulate obtained is dry mixed with 12.5 g. NAD, 3.75 g. MTT, 0.75 g. PMS and 80 g. urethane. The mixture is pressed to form reagent tablets of 12 mm. diameter which contain, per tablet, 12.5 mg. NAD, 3.75 mg. MTT and 0.75 mg. PMS. The urethane is sublimated from these tablets for 8 hours in a freeze drying plant at 0°C. and 10^{-1} or 10^{-3} mm.Hg.

The results of tests carried out on these tablets, in the manner described in Example 1, are set out in the following Table 3:—

TABLE 3

tablet	height (mm.)	hardness (kg.)	dissolving time (sec.)	breakability (sec.)
A	3.5	12	660	540
B	4.2	3	480	15

Example 4.

Tablet A: 500g. sodium chloride are ground, mixed with 116 g. sodium *p*-nitrophenyl phosphate, precompressed and sieved. There are pressed tablets of 5 mm. diameter containing 116 mg. sodium *p*-nitrophenyl phosphate.

Tablet B: 500 g. sodium chloride are ground, mixed with 116 g. sodium *p*-nitrophenyl phosphate and 134 g. urethane, precompressed and sieved. There are pressed tablets of 5 mm diameter containing 11.6 mg. sodium *p*-nitrophenyl phosphate. These tablets are heated for 10 hours in a drying cabinet at 30°C. to sublimate the urethane.

The results of tests carried out on these tablets, in the manner described in Example 1, are set out in the following Table 4:

TABLE 4

tablet	height (mm.)	hardness (kg.)	dissolving time (sec.)	breakability (sec.)
A	1.9	3	300	60
B	2.4	1	120	<10

WHAT WE CLAIM IS:—

1. A process for the production of porous tablets, wherein the tablet components are hard pressed together with at least one inert solid adjuvant which sublimes at a temperature which does not adversely affect any of the tablet components, whereafter said adjuvant is sublimated.

2. A process according to claim 1, wherein the adjuvant is sublimated in a vacuum.

3. A process according to claim 1 or 2, wherein the adjuvant used is urethane, urea, ammonium carbonate, ammonium bicarbonate, hexamethylene-tetramine, benzoic acid, phthalic anhydride, naphthalene or camphor.

4. A process according to any of the preceding claims, wherein the amount of adjuvant used is 5—50% by weight, referred to the total tablet mass.

5. A process according to claim 4, wherein the amount of adjuvant used is 10 to 30% by weight, referred to the total tablet mass.

6. A process according to any of the preceding claims, wherein the tablets additionally contain conventional water-soluble carrier materials.

7. Process according to claim 1 for the production of porous tablets, substantially as hereinbefore described and exemplified.

8. Porous tablets, whenever produced by the process according to any of claims 1 to 7.

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(54) ALKANESULPHONATE SPRAY PRODUCT

(71) We, HOECHST AKTIENGESELLSCHAFT, a Body Corporate organised under the laws of the Federal Republic of Germany of 6230 Frankfurt/Main 80 Germany,

do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a spray product containing an alkali metal alkanesulphonate as surfactant and to a process for its preparation.

It has been proposed to prepare detergent powders by spray drying mixtures of active detergent substances, e.g. soaps, alkarylsulphonates, alkenesulphonates and alkanesulphonates, together with other predominantly inorganic additives as carriers. Whereas spray drying of mixtures of the first three above-mentioned detergent substances and inorganic additives generally yields free-flowing, pourable powders which do not cake together on storing, the products of spray drying mixtures of alkanesulphonates and inorganic additives generally have poor storage properties if the detergent content is high. Since alkanesulphonates are hygroscopic, spray products produced therefrom with a high content of alkanesulphonates tend to cake or even become sticky as a result of water absorption from the atmosphere or from the inorganic additives which contain water of crystallisation. For this reason alkanesulphonates have hitherto been used satisfactorily only in solution e.g. in liquid cleaning materials and bath additives.

There have been numerous attempts to overcome the problem of the poor storage properties of alkanesulphonates and to facilitate the preparation of spray products containing alkanesulphonates by adding small quantities of so-called "anti-caking agents" to the spray mixture. Thus benzenesulphonates, toluenesulphonates and magnesium silicate have for example been proposed as "anti-caking agents". In addition, high-melting, finely divided powders which are not hygroscopic have been added to the spray-dried products. Such powders act purely mechanically by coating the individual granules of the spray product, thus preventing them from sticking to one another. It is also possible to reduce caking on storage by greatly increasing the proportion of carrier to detergent but this is commercially undesirable in that the detergent strength of the powder is reduced.

According to the present invention we now present a free-flowing spray-dried powder comprising A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate being at least 15%, preferably at least 20% by weight referred to the combined weight of carrier and alkanesulphonate and and the content of silica being from 0.2 to 6%, preferably from 0.3 to 4% by weight referred to the weight of alkanesulphonate.

The spray-dried powders according to the invention are in general free-flowing, non-hygroscopic and thus non-caking. They may be prepared by spray-drying an aqueous composition containing A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate in the composition being at least 15%, preferably at least 20% by weight referred to the combined weight of the carrier and alkanesulphonate and the content of silica being from 0.2 to 6%, preferably from 0.3 to 4% by weight referred to the weight of alkanesulphonate at a temperature from 100 to 350°C, preferably from 120 to 320°C,

which process constitutes a further feature of the invention.

The alkanesulphonate component of the spray-dried powders according to the invention preferably comprises alkanesulphonates containing from 12 to 18 carbon atoms in the alkyl group and the alkali metal is preferably sodium or potassium.

The silica is conveniently added to the aqueous spray composition in the form of a dispersion having a bulk weight of from 60 to 400g per litre.

The carrier generally should have a melting point of at least 150°C. Salts of non-corrosive acids are preferred, particularly their sodium salts such as sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate and sodium citrate. Small quantities of sodium chloride may also be contained in the spray mixture, for example as impurity introduced into the above-named sodium salts during their preparation. Other suitable carrier salts include for example potassium and ammonium sulphate, potassium carbonate and a potassium and an ammonium polyphosphate. Mixtures of carrier salts may also be used. The carrier is preferably soluble in water at 100°C in an amount of at least 30% by weight.

The spray-dried powder may if desired contain other additives conventionally used in washing powders, for example optical brighteners, bleaches and soaps.

The process of preparing the spray-dried powder according to the invention may be effected in a conventional spray plant. Thus for example the hot aqueous spray composition may be atomised by passage through a nozzle and brought into contact with a flow or counterflow of hot gases. The aqueous composition is preferably as concentrated as possible for the spray-drying method. Water contained in the spray composition evaporates and a spray product is obtained which consists of approximately spherical, non-caking particles. The bulk weight and water content of the spray product may be adjusted by selecting appropriate spray conditions, the spray temperature being particularly important.

The spray-dried powders according to the invention may be used as components of surfactant compositions optionally in admixture with the aforementioned additional additives. They may also be used as lubricants or carriers is cosmetic preparations. It is also possible to use the process according to the invention for a one-step preparation of commercial detergent compositions.

The following Examples serve to illustrate the preparation of spray-dried powders according to the invention.

Example 1

A 35% aqueous spray solution containing a mixture of sodium sulphate and sodium alkanesulphonates (containing, on average, 15 carbon atoms in the alkyl group) in a weight ratio of 1.5:1 is prepared. 4% by weight (based on the weight of alkanesulphonates) of a fine dispersion of silica with a bulk weight of 100g per litre is dispersed in the aqueous spray solution. The mixture is then sprayed at a temperature between 315 and 340°C. A white, free-flowing, non hygroscopic spray-dried powder with a water content of about 7% by weight and a bulk weight of 300 to 400g per litre and which does not cake on storage is obtained.

Example 2

A 32% aqueous spray solution containing a mixture of sodium alkanesulphonates containing 12 to 18 carbon atoms in the alkyl groups and sodium tripolyphosphate in a weight ratio of alkylsulphonates to tripolyphosphate of 1:3 is prepared. 0.375% by weight (based on the weight of alkanesulphonates) of a fine silica dispersion with a bulk weight of 100g per litre is dispersed therein. The mixture is sprayed as in Example 1 to yield a white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 300 to 400g per litre and a water content of 10% by weight and which does not cake on storage.

Example 3

A 45% aqueous spray solution containing a mixture of sodium alkanesulphonates containing predominantly alkanesulphonates with 12 to 15 carbon atoms, sodium sulphate and sodium tripolyphosphate in a weight ratio of alkanesulphonates to sulphate to tripolyphosphate of 1:2:2 is prepared. 1.1% by weight (based on the weight of alkanesulphonates) of a fine silica dispersion with a bulk weight of 200g per litre is dispersed therein. The mixture is sprayed as in Example 1 to yield a white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 180 to 250g per litre and a water content of 7% by weight and which does not cake on storage.

Example 4

A spray-dried powder is prepared analogously to Example 1 except that the aqueous spray mixture is sprayed at a temperature of 290 to 300°C. A white, free-flowing, non-hygroscopic spray-dried powder with a bulk weight of 300 to 400g per litre and a water content of about 8% by weight and which does not cake on storage is obtained.

Comparison Example A

Spray products are prepared analogously to Examples 1 to 2 but omitting the silica dispersion. The products are white and initially free-flowing but cake on storage.

Comparison Example B

A 33% aqueous spray solution containing

a mixture of sodium alkanesulphonates containing 12 to 18 carbon atoms in the alkyl groups and sodium tripolyphosphate in a weight ratio of alkanesulphonates to tripolyphosphate of 2:15 and containing no silica dispersion is sprayed analogously to Example 1 to yield a white, free-flowing product which does not cake on storage and which has a bulk weight of 300 to 400g per litre and a water content of 15% by weight.

Comparison Example C

A spray-dried powder is prepared analogously to comparison Example B except that tetrasodium pyrophosphate is used in the place of sodium tripolyphosphate. A white, free-flowing product which does not cake on storage and which has a bulk weight of 300 to 400g per litre and a water content of 15% by weight is obtained.

WHAT WE CLAIM IS:—

1. A free-flowing spray-dried powder comprising A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate being at least 15% by weight referred to the combined weight of carrier and alkanesulphonate and the content of silica being from 0.2 to 6% by weight referred to the weight of alkanesulphonate.

2. A spray-dried powder as claimed in claim 1 wherein the content of silica is from is from 0.3 to 4% by weight referred to the weight of alkanesulphonate.

3. A spray-dried powder as claimed in claim 1 or claim 2 wherein the carrier has a melting point of at least 150°C.

4. A spray-dried powder as claimed in any of the preceding claims wherein the carrier is sodium carbonate, sodium metasilicate, sodium tripolyphosphate, sodium sulphate, sodium citrate, potassium carbonate, a potassium polyphosphate, potassium sulphate, ammonium sulphate or an ammonium polyphosphate.

5. A spray-dried powder as claimed in claim 3 or claim 4 wherein the carrier is soluble in water at 100°C in an amount of at least 30% by weight.

6. A spray-dried powder as claimed in any of the preceding claims wherein the alkanesulphonate component comprises an alkanesulphonate containing from 12 to 18 carbon atoms.

7. A spray-dried powder as claimed in any of the preceding claims wherein the alkanesulphonate component comprises a sodium or potassium alkanesulphonate.

8. A spray-dried powder as claimed in any of the preceding claims wherein the content of alkanesulphonate is at least 20% by weight referred to the combined weight of carrier and alkanesulphonate.

9. A spray-dried powder as claimed in any of the preceding claims which additionally contains at least one further ingredient selected from optical brightening agent, bleaches and soaps.

10. A spray-dried powder as defined in claim 1 substantially as herein disclosed.

11. A spray-dried powder as defined in claim 1 substantially as herein disclosed. with particular reference to Example 1 to 3.

12. A spray-dried powder as defined in claim 1 substantially as herein described with particular reference to Example 1 to 3.

13. A process for the preparation of a free-flowing spray-dried powder containing an alkali metal alkanesulphonate as surfactant which comprises spray-drying at a temperature of from 100 to 350°C an aqueous composition containing A) a carrier comprising at least one salt of an inorganic or organic acid and an inorganic base, B) at least one alkali metal alkanesulphonate as surfactant and C) finely dispersed silica, the content of alkanesulphonate in the composition being at least 15% by weight referred to the combined weight of the carrier and alkanesulphonate and the content of silica being from 0.2 to 6% by weight referred to the weight of alkanesulphonate.

14. A process as claimed in claim 13 wherein the composition is spray dried at a temperature of from 120 to 320°C.

15. A process as claimed in claim 13 or claim 14 wherein the content of silica in the composition is from 0.3 to 4% by weight.

16. A process as claimed in any of claims 13 to 15 wherein the silica is introduced into the aqueous composition in the form of a fine dispersion having a bulk weight of 60 to 400g per litre.

17. A process for the preparation of a spray-dried powder according to claim 13 substantially as herein described.

18. A process for the preparation of a spray-dried powder according to claim 13 substantially as herein described with reference to Examples 1 to 4.

19. A process for the preparation of spray-dried powder according to claim 13 substantially as herein described with reference to Examples 1 to 3.

20. A free-flowing spray-dried powder whenever prepared by a process as claimed in any of claims 13 to 18.

21. A surfactant composition comprising a free-flowing spray-dried powder as claimed in any of claims 1 to 12 and 20.

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